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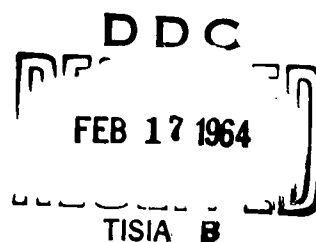
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**DIELECTRIC MATERIALS
FOR
HIGH TEMPERATURE EVALUATION**

Report Period: 15 September to 16 December 1963

Report RMD 5038-Q-1
Contract No. NObs 90065
Index No SR-007-03-03
Task No. 1000



Thiokol®
CHEMICAL CORPORATION
REACTION MOTORS DIVISION
DENVER, NEW JERSEY

DIELECTRIC MATERIALS FOR HIGH TEMPERATURE EVALUATION

Joseph Green
Nathan Mayes


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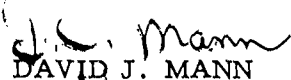
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ABSTRACT

Research was conducted toward the preparation of thermally stable carborane siloxane materials for use as embedding compounds and elastomers. Hydrocyclosiloxanes were prepared for subsequent conversion to the corresponding carborane cyclic compounds. A carborane cyclotetra~~siloxane~~ siloxane was prepared and polymerized.

A linear carborane siloxane of $> 7,000$ molecular weight was prepared and its thermal properties evaluated by DTA and TGA.

ADMINISTRATIVE INFORMATION

Research efforts to prepare thermally stable dielectric materials and elastomers were originally authorized by Bureau of Ships Contract NObs 84774, 15 June 1961. The Navy Index Number was SR-007-03-03, Task 1000. That contract expired 15 December 1962. Technical work was resumed 15 February 1963 under Bureau of Ships Contract NObs 88318, Index Number SF-007-03-03, Tasks 1000 and 1003 and continued through 1 September 1963. Technical work was again resumed 15 September 1963 under Contract NObs 90065, Index No. SR-007-03-03, Task 1000, RMD Project 5038.

The Bureau of Ships Project Engineer is Mr. William Shetterly. The Project Supervisor at Thiokol Chemical Corporation, Reaction Motors Division is Mr. Joseph Green. Other contributing personnel include Mr. A. Jackson, Mr. A. Lum, Mr. N. Mayes, Mr. R. Michaels and Mr. J. Paustian.

SUMMARY

THE PROBLEM

The program objectives are to prepare carborane-based compounds useful as dielectric materials over the range of 25 to 500°C and prepare carborane based elastomers useful over the same range.

FINDINGS

A 50 gallon Pfaudler jacketed glass-lined reactor was used to prepare bromomethylcarborane on a pilot plant scale. The reaction was based upon a 30 pound charge of decaborane, 192 pounds of acetonitrile and 37.2 pounds of propargyl bromide to yield 38.5 pounds of crude product.

Conversion of the above bromomethylcarborane to butenylcarborane is in progress.

A carborane cyclotetrasiloxane was prepared and polymerized.

Linear carborane siloxanes of > 7,000 molecular weight were obtained without catalyst.

DTA of carborane silicone indicated no thermal reaction prior to decomposition at 460°C.

I. BACKGROUND

Several years ago, Thiokol-RMD demonstrated the outstanding thermal stability of the carborane nucleus (Ref 1). On the basis of this work, a contract was awarded by the Bureau of Ships to Thiokol Chemical Corporation, Reaction Motors Division, for the preparation and evaluation of prototype carboranes as model compounds for ultrahigh temperature dielectric materials (Ref 2). The thermal stability and dielectric results obtained on the initial contract (Ref 3) were sufficiently promising so that a continuation contract was awarded in which the goals were:

1. To study and develop thermally stable carborane-based dielectric embedding materials.
2. To study and develop carborane-based elastomers for high temperature application.

The target characteristics for the embedding materials were:

- Should be suitable for embedding electronic parts capable of continuous operation at 350°C and 3,000 hours at 500°C.
- Should have minimum shrinkage and be nonporous.
- Should closely match metals in thermal expansion and be capable of withstanding thermal shock.
- Should have the following electrical characteristics over the temperature range, ambient to 500°C.
 - Volume resistivity of 10^{10} ohm-cm (min)
 - Dissipation factor of 0.07 (max)
 - Dielectric constant of 5.0 (max) at frequencies of 1 mc and 100 mc
 - Dielectric strength of 1000 volts/mil at 60 cps.

- Should have a volume resistivity of 10^{13} ohm-cm (min) at 23°C after 230 hours at 70°C suspended over water in a sealed container.
- Should be self-extinguishing when tested as specified in Military Specification MIL-I-16823.

The required characteristics of the thermally stable elastomers were:

- Tensile strength of the vulcanized elastomer should be greater than 600 psi when tested at ambient temperature up to 500°C .
- Ultimate elongation of the vulcanized rubber should be not less than 200 percent when tested at 500°C .
- Shore A durometer hardness should not change more than 20 points after the elastomer has been subjected to oven aging at 500°C for 300 hours.
- Vulcanizates produced from the carborane gum elastomers should have the following initial properties:

Tensile Strength, min	1500 psi
Ultimate Elongation, min	300%
Hardness (Shore A)	between 40 and 80.

- Vulcanizates should not be affected appreciably by immersion in water at 212°F and should not absorb more than one percent of its weight in water when immersed for 30 days at $75 \pm 3^{\circ}\text{F}$.

Under the continuation contract (NObs 84774), a number of carborane-based materials were synthesized and evaluated for thermal stability and electrical properties. They included carborane-fluorocarbon polyesters, carborane-epoxies, carborane-phenylenes and carborane-silicones. Of these, the polyesters did not demonstrate the required thermal stability, and the epoxies and phenylene derivatives, although promising on the basis of thermal properties, presented difficulties in synthesis that removed them from consideration except as a long range research effort. The silicone derivatives

showed excellent promise with electrical properties that met the program specifications and thermal stability at temperatures greater than 400°C. Work was therefore concentrated in this area, and the program was reoriented into three phases: (1) improvement of the process for monomer and polymer synthesis to allow for early scaleup, (2) studies leading to higher molecular weight polymers for elastomer and embedding compound applications and (3) curing studies.

By the end of the contract period, significant progress had been made in each phase. Process development studies resulted in greatly improved yields and shorter reaction times. For example, starting with bromomethylcarborane, the overall yield of the monomer, (4-carboranylbutyl) methyldichlorosilane, was increased from 8 to 52 percent, and the preparative time was substantially reduced. Changing the method of polymer preparation from catalyzed condensation to cohydrolysis reduced polymerization time from five days to two hours. In the second area, polymer molecular weights of ca. 2,000 obtained initially were increased to ca. 13,000, and progress was made in isolating cyclosiloxanes through which polymers of much higher molecular weight may ultimately be prepared. Initial curing studies indicated that the liquid polymer could be cured through reactive end groups and showed that the carborane silicones could be cured in the several ways common in silicone polymer technology.

A continuation contract (NObs 88318) was awarded on the basis of the previous results and the potential ability of carborane siloxanes to meet the goals of the program. The goals and target requirements have not changed. Greater emphasis has been placed on the development and fabrication of end items. Thus, the preparation of carborane cyclosiloxanes as intermediates for high molecular weight siloxanes, the preparation and curing of multifunctional carborane silicones, the preparation of carborane siloxane liquid plasticizers, the preparation of fiber glass cloth laminates, and studies of the adhesion of carborane siloxanes to metals were investigated.

The present contract (NObs 90065) continues the investigation and development of carborane siloxanes for high temperature service. The immediate objectives are:

- Prepare 20 to 30 pounds of carborane silicone.
- Conduct curing studies.
- Prepare glass-resin laminates for physical, thermal and electrical testing.
- Prepare experimental varnishes for evaluation of high temperature resistant electrical wire coatings.

In the existing process, the acetonitrile was stripped completely from the reaction mixture and the oily residue was treated with aqueous caustic to eliminate non-carborane B-H. This reaction is exothermic and evolves hydrogen. Although no difficulty was experienced on a small scale, it was expected that it would be difficult to control on a large scale and that reaction would be incomplete.

After separation of the caustic layer (and washing to neutrality), the product was extracted from the residue with pentane or hexane. Because of the viscous nature of the residue, it was thought that extraction on a large scale would be tedious and inefficient, and would result in a large loss of product. Therefore, the process was modified to include addition of a higher boiling solvent (toluene) to the reaction mixture during the acetonitrile strip. This resulted in more complete acetonitrile removal and a fluid residue. Thus, heat transfer and interfacial contact during the caustic treatment was greatly increased, resulting in a controllable, efficient reaction. Furthermore, no extraction step was required since the bromomethylcarborane was already in solution.

As an example of the increase in efficiency resulting from this change, one reaction (2.0 mole, 245 g $B_{10}H_{14}$ charged) was completed with a yield of crude bromomethylcarborane of 394 gm, 83.3% of theory. Control reactions run at 0.2 and 2.0 molar scale, but with the original process, resulted in yields of only 19.0 and 207 gm, 40.0 and 43.8% of theory. One additional reaction was initiated to determine processability at bench-scale (1980 gm, 16.2 moles of $B_{10}H_{14}$). This reaction was not completed to the point of product isolation because of time and space limitations; however, the improvements in processability were confirmed.

At this point, it was believed that a reaction of pilot plant-scale could be successfully effected and a reaction was designed to furnish the quantity of bromomethylcarborane required for the program. This reaction was based upon a 30 pound charge of decaborane, 192 pounds of acetonitrile and 37.2 pounds of propargyl bromide. A 50 gallon Pfaudler jacketed, glass-lined reactor with variable-speed agitator, heat exchanger, and receiver was used. A flow sheet of the process is shown as Figure 1. This preparation was successful and 38.5 lb of crude bromomethylcarborane was obtained. Methods of purification of this material to the degree required for conversion to butenylcarborane are now being investigated.

The preparation of butenylcarborane for conversion to the silane monomer and for the determination of process improvements was also continued. In

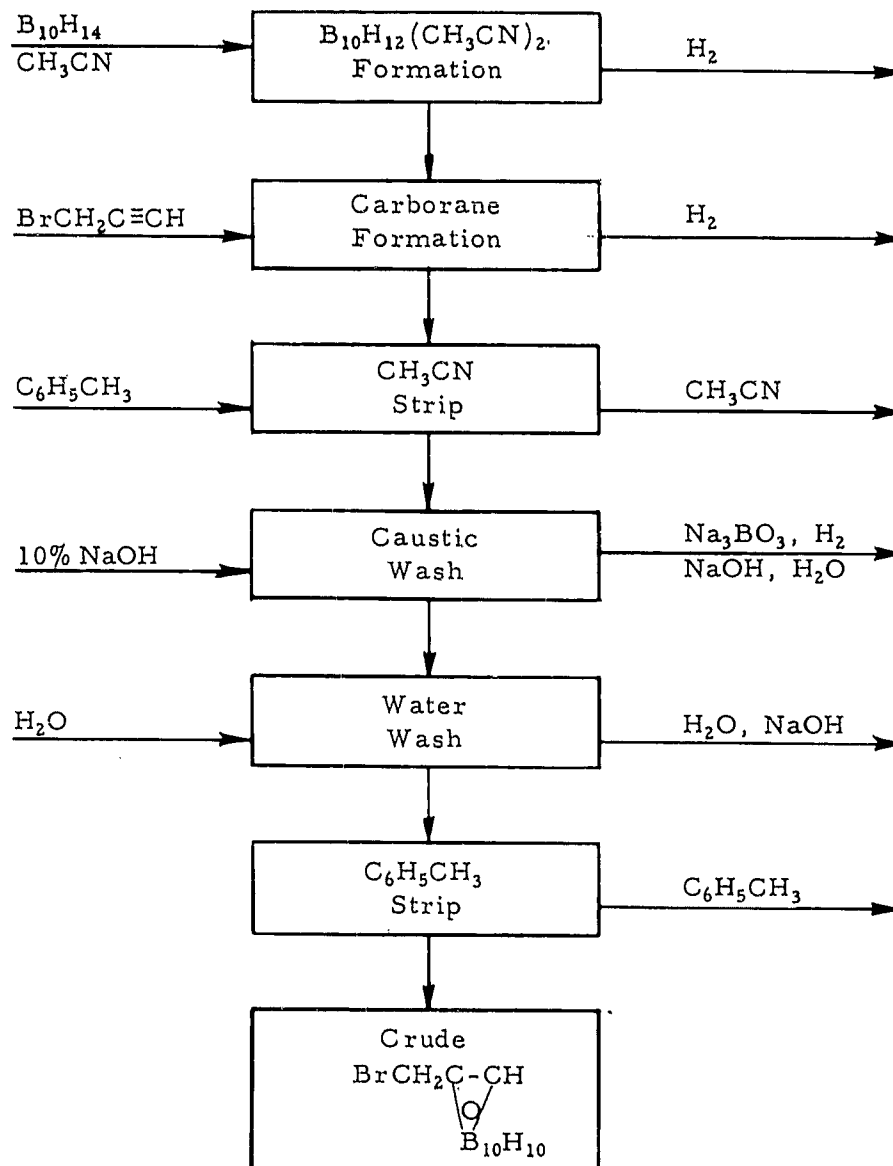
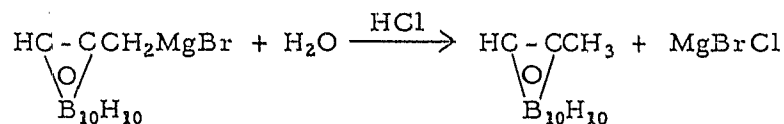


Figure 1. Flowsheet for Bromomethylcarborane Formation

this reaction, some problem areas were also anticipated as the result of operations at a larger scale. Previously, the relatively small quantities of bromomethylcarborane had been purified by rectification at less than 2 Torr. This degree of vacuum is more difficult to maintain at a large scale, but, more important, the fact that pure bromomethylcarborane is a solid at room temperature makes any vacuum distillation difficult. Plugging in the column, the condenser, and stopcocks is a continuing problem. Therefore, methods of purification other than distillation need to be investigated.

Modifications of the butenylcarborane reaction may also be necessary. For example, relatively large volumes of ether have been used. If the ether requirements could be reduced without affecting product yields and purity, more product could be obtained per batch. More important, however, is the quantity of methylcarborane produced as a by-product due to hydrolysis of unreacted carboranylmethylmagnesium bromide.



The methylcarborane was previously removed by distillation, but since it is a high-melting solid, severe mechanical problems are expected on a larger scale operation.

Two reactions were completed to determine the feasibility of using the bromomethylcarborane as isolated and without further purification. In both reactions, large amounts of methylcarborane were formed (in one reaction, about 50 percent). Although this is not absolute proof that "as isolated" bromomethylcarborane cannot be used, additional reactions will be made with purified product. One reaction is now in progress utilizing bromomethylcarborane purified by treatment with methanol (to remove the last traces of non-carborane B-H), followed by solution in hexane with filtration of insolubles (e.g., boric acid).

Methods for the minimization of methylcarborane formation which are being investigated include treatment of the reaction mixture (after coupling with allyl bromide) with materials which could be expected to react with unreacted carboranylmethylmagnesium bromide, but which after hydrolysis, would yield more easily separable products. These materials include CO₂ to

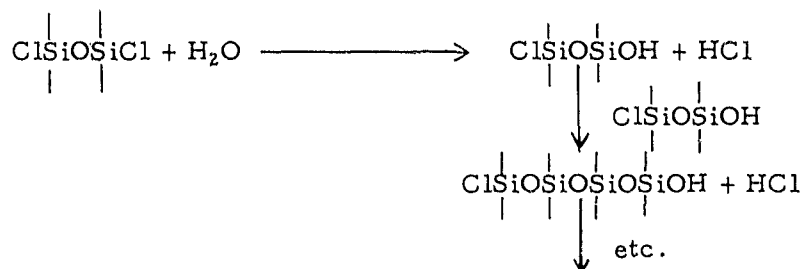
form $\text{HC} \begin{array}{c} \diagup \text{O} \diagdown \\ \text{B}_{10}\text{H}_{10} \end{array} \text{COOH}$ separable as the water soluble salt, and such compounds

as benzyl bromide to yield coupled products which should be much less volatile than butenylcarborane. The results of these reactions will be reported when complete.

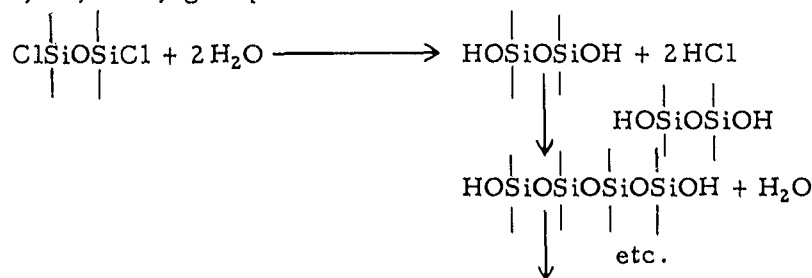
B. Preparation of "Alternating" Carborane Siloxane Polymers

Polymer was prepared by the hydrolysis of 1,1,3-trimethyl-3-(4-carboranylbutyl)-1,3-dichlorodisiloxane. The polymer is believed to have a degree of structural regularity and it may be obtained free of catalyst residues and free of cyclic products (Ref 4). The most recent material prepared had a molecular weight of 7,200 after solvent fractionation. Samples were subjected to DTA and TGA measurements (cf. Phase II) and a sample has been submitted to USNASL for thermal evaluation. USNASL will also evaluate the suitability of this siloxane for internal motor application at elevated temperatures.

Previously the simple hydrolysis of carborane silanes without catalyst yielded products of considerably lower molecular weight (ca. 2,000). The reason that higher polymers may be obtained by the hydrolysis of 1,1,3-trimethyl-3-(4-carboranylbutyl)-1,3-dichlorodisiloxane is apparently the way the hydrolysis is done rather than any greater reactivity of the dichlorodisiloxane monomer. If one mole of water is added per mole of dichlorodisiloxane the polymerization reaction is essentially one of condensation of silyl hydroxy groups with silyl chloride groups.



If two moles of water are added then the polymerization involves condensation of silyl hydroxy groups.

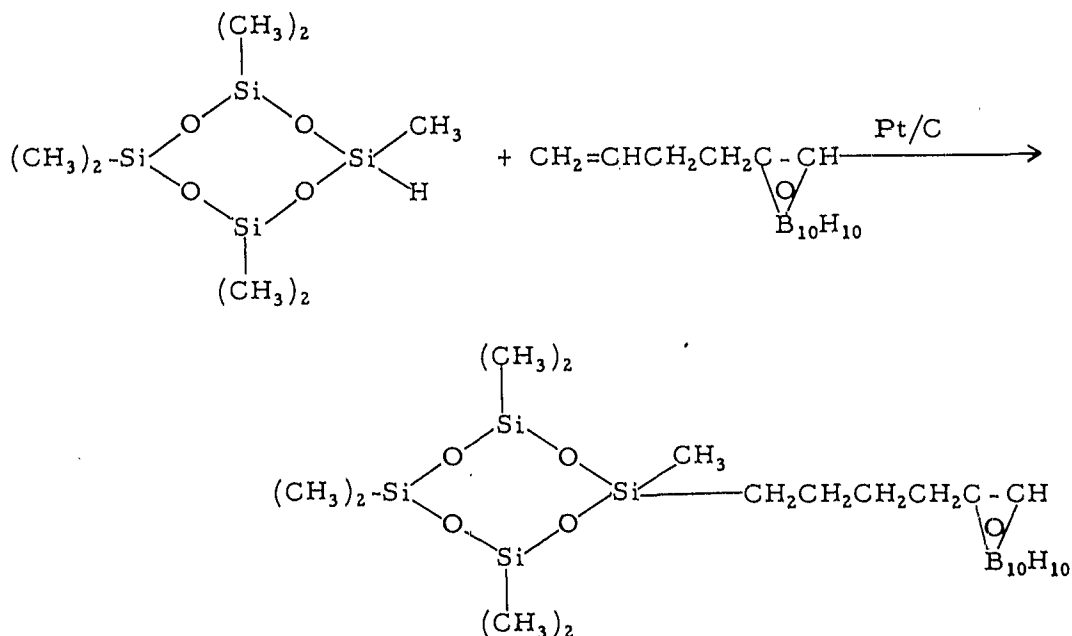


The hydroxy condensation occurs readily in the case of dimethylsilicones without catalyst but bulky substituents such as phenyl or carborane greatly reduce hydroxy reactivity and make catalysts necessary for the attainment of high molecular weight.

C. Higher Molecular Weight Polymers Through Cyclosiloxanes

The preparation of hydrocyclosiloxane trimers and tetramers has been reported previously (Ref 5). The tetramers were obtained directly from the cohydrolysis of dimethyldichlorosilane and methyl dichlorosilane. The trimers were obtained by pyrolysis of the higher molecular weight cohydrolysis products. None of these materials had been isolated due to the difficult fractional distillation necessary. The materials have now been fractionally distilled through a Heli-grid column of 60 theoretical plates. The physical constants of these products closely match literature values or their chemical analysis is close to the theoretical values. The hydrocyclosiloxanes that have been obtained are shown in Table I with pertinent data. The trimers are new compounds and no literature values are available.

A carborane derivative was prepared from the monohydrocycloctetrasiloxane and subjected to polymerization studies using acid and base catalysis.



bp 148-156°C/5 × 10⁻⁵ mm; n_D²⁰ 1.4780; mp 35-43°C.

TABLE I
PHYSICAL CONSTANTS OF HYDROCYCLOSILOXANES

Product	bp, °C/50 mm Hg		$\frac{n_D^{20}}$		$\frac{\%H}{\text{Calculated}}$	
	Found	Literature (Ref 7)	Found	Literature (Ref 6)	Found	Calculated
$-\text{[OSiCH}_3\text{(H)]}_4-$	57		1.3857	1.3870		
$-\text{[OSiCH}_3\text{(H)]}_3-\text{[OSi(CH}_3)_2]$	69	69	1.3900	1.3900		
$-\text{[OSiCH}_3\text{(H)]}_2-\text{[OSi(CH}_3)_2]_2-$	78	77.5	1.3920	1.3927		
$-\text{[OSiCH}_3\text{(H)]}-\text{[OSi(CH}_3)_2]_3-$	85	84.5	1.3943	1.3950		
$-\text{[OSiCH}_3\text{(H)]}_2-\text{[OSi(CH}_3)_2]$	36.5		1.3797		1.11	1.04
$-\text{[OSiCH}_3\text{(H)]}-\text{[OSi(CH}_3)_2]_2-$	47.3		1.3823		0.511	0.483

In the presence of 0.34% sulfuric acid, polymerization to a clear, colorless, very viscous liquid of molecular weight 1420 occurred. Treatment of the material with potassium hydroxide resulted in oxidation and gelation when heated in air at 170°C and no reaction when heated in an evacuated vessel at 100°C.

PHASE II. THERMAL EVALUATION

Previous thermal studies on carborane silicone polymer carried out using TGA and isoteniscope techniques have indicated that catastrophic decomposition occurs at ca. 460°C. These studies have failed to determine, however, if there is a rearrangement or equilibrium of linear polymer to cyclic at some lower temperature as occurs with dimethylsilicones. If carborane cyclosiloxanes did form they would not be volatile at these temperatures and would not effect a weight change in TGA or a pressure increase in the isoteniscope. In order to more completely determine the thermal characteristics of the carborane silicones, a specially prepared polymer sample (cf. Phase I) was subjected to differential thermal analysis (DTA) as well as TGA. The results are shown in Figures 2 and 3. It is seen from the DTA that no thermal reaction occurs previous to the catastrophic decomposition at 460°C. The TGA also shows catastrophic decomposition in this region with most rapid weight loss occurring at above 450°C. This material is also undergoing thermal evaluation at USNASL.

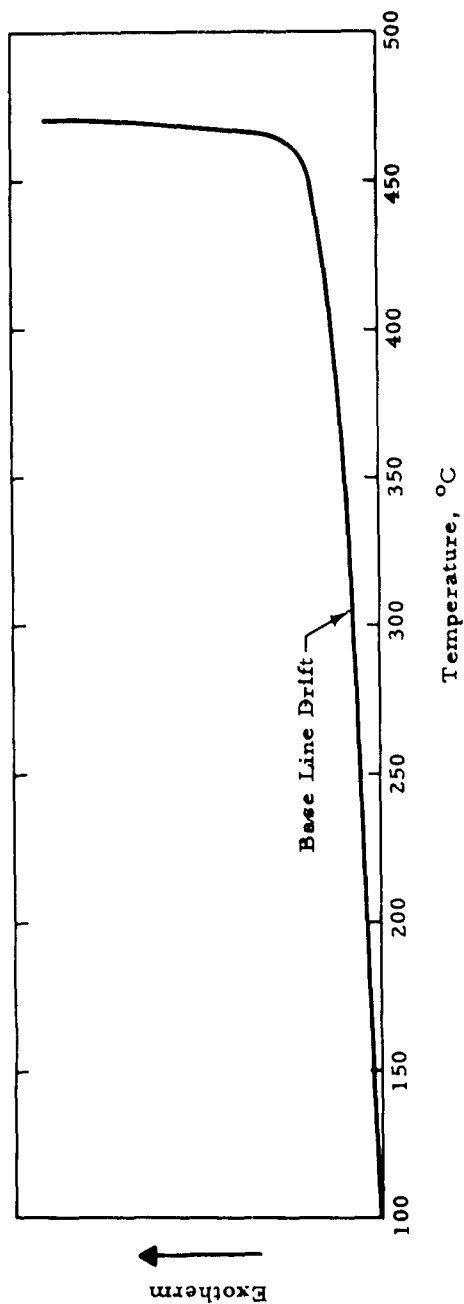


Figure 2. DTA of Carborane Siloxane (Sample 167389) Under Nitrogen

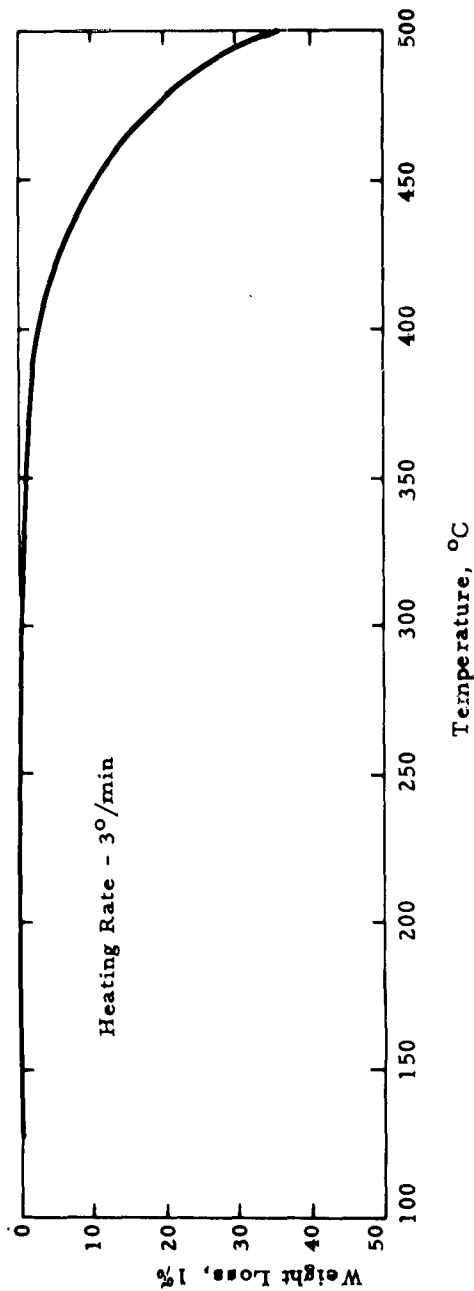


Figure 3. TGA of Carborane Siloxane (Sample 167389) Under Nitrogen

III. CONCLUSIONS AND RECOMMENDATIONS

A. ACCOMPLISHMENTS AND CONCLUSIONS

The scaleup preparation of carborane silicones has been initiated with the preparation of bromomethylcarborane on a pilot plant scale. The reaction was based upon a 30 pound charge of decaborane, 192 pounds of acetonitrile and 37.2 pounds of propargyl bromide to yield 38.5 pounds of crude product. Conversion to butenylcarborane is in progress.

The preparation and separation by fractional distillation of hydrocyclosiloxanes proved to be a major task due to the difficulties of separation. However, tetramers containing 4, 3, 2 and 1 hydrogens have been isolated and trimers containing 2 and 1 hydrogens have been obtained. The latter compounds are materials previously unreported in the literature.

A cyclotetrasiloxane containing one butylcarborane substituent was prepared and preliminary polymerization studies were done. This cyclic, which is the least substituted of the class, should be the most reactive toward polymerization since bulky substituents tend to inhibit ring opening. The preliminary studies indicated that this compound does polymerize but reactivity is greatly reduced by the single butylcarborane substituent. Other factors which may have a bearing on the polymerization such as catalyst concentration, type of catalyst, temperature, and solvent have not been studied; however, it appears desirable to use cyclotrisiloxanes in order to obtain high reactivity toward polymerization. The higher reactivity of the trimers is illustrated by the methylcyclosiloxanes where the rate of polymerization of the trimer exceeds that of the tetramer by a factor of 354.

The DTA of carborane siloxane has indicated no thermal reactions prior to complete decomposition. Some further experiments are necessary to conclusively show that thermal rearrangements do not occur. If the polymer maintains constant molecular weight, on being heated at temperatures below 460°C, such rearrangements may be ruled out. DTA should be of value in determining oxidative stability, by indicating at what temperature reaction

with oxygen occurs. Further experiments in an oxygen atmosphere to determine weight loss or gain, viscosity change or gelation, and products of oxidation may also be made to determine oxidative stability.

B. RECOMMENDATIONS

The following work outline is recommended for the remainder of the program:

- Continue scaled up polymer preparation.
- Develop curing procedures and prepare fiber glass laminates and varnishes.
- Study the equilibrium or rearrangement of linear polymer.
- Determine oxidative stability.

IV. REFERENCES

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2. Contract NObs-78416, Index No. SR-007.
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<p>THIokol CHEMICAL CORPORATION, REACTION MOTORS DIVISION, DENVER, N. J., DIELECTRIC MATERIALS FOR HIGH TEMPERATURE EVALUATION, by Joseph Green and Nathan Mayes. December 1963, 15 p. incl. figs., tables and refs. (Index No. SR-007-03-03, Task 1000) (Contract No. NObs 90065)</p> <p>Research was conducted toward the preparation of thermally stable carborane siloxane materials for use as embedding compounds and elastomers. Hydrocyclosiloxanes were prepared for subsequent conversion to the corresponding carborane cyclic compounds. A carborane cyclotetrasiloxane was prepared and polymerized.</p> <p>A linear carborane siloxane of > 7,000 molecular weight was prepared and its thermal properties evaluated by DTA and TGA.</p>	<p>UNCLASSIFIED</p> <p>1. Preparation of Thermally Stable Materials</p> <p>2. Thermal Evaluation</p> <p>I. Contract NObs 90065</p>	<p>THIokol CHEMICAL CORPORATION, REACTION MOTORS DIVISION, DENVER, N. J., DIELECTRIC MATERIALS FOR HIGH TEMPERATURE EVALUATION, by Joseph Green and Nathan Mayes. December 1963, 15 p. incl. figs., tables and refs. (Index No. SR-007-03-03, Task 1000) (Contract No. NObs 90065)</p> <p>Research was conducted toward the preparation of thermally stable carborane siloxane materials for use as embedding compounds and elastomers. Hydrocyclosiloxanes were prepared for subsequent conversion to the corresponding carborane cyclic compounds. A carborane cyclotetrasiloxane was prepared and polymerized.</p> <p>A linear carborane siloxane of > 7,000 molecular weight was prepared and its thermal properties evaluated by DTA and TGA.</p>	<p>UNCLASSIFIED</p> <p>1. Preparation of Thermally Stable Materials</p> <p>2. Thermal Evaluation</p> <p>I. Contract NObs 90065</p>
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